Nitrogen Doping of ZnSe and CdTe Epilayers: A Comparison of Two rf Sources

M. MOLDOVAN,[1] L.S. HIRSCH,[1] A.J. PTAK,[1] C.D. STINESPRING,[2] T.H. MYERS,[1] and N.C. GILES[1,3]

1.—Department of Physics, West Virginia University, Morgantown, WV 26506. 2.—Department of Chemical Engineering, West Virginia University, Morgantown, WV 26506. 3.—e-mail: ngiles@wvu.edu

Nitrogen is the most promising dopant for p-type ZnSe, and is attractive for CdTe p-type doping, but unwanted compensating centers have limited the maximum achievable carrier densities in both of these material systems. One important factor is the nitrogen source. The characteristics of the nitrogen source (i.e., ratio of charged to neutral species) influence both the nitrogen incorporation rate and the types of centers which may be produced in epilayers. We have performed a study of ZnSe:N and CdTe:N using two different rf plasma sources: an Oxford CARS-25 and an EPI Unibulb. The EPI source has a very high efficiency for producing atomic nitrogen (~70%) while maintaining a very low total ion content. Both sources gave a high incorporation of nitrogen; however, Hall measurements indicate that compensation is still a problem in p-type doping of these materials. A detailed liquid-helium-temperature photoluminescence study has been performed to monitor changes in the defect structure produced using different source operating parameters.

Key words: CdTe:N, nitrogen doping, p-type doping, photoluminescence, ZnSe:N

INTRODUCTION

P-type doping of CdTe and especially ZnSe has proven to be difficult. Nitrogen is currently the most promising candidate. Incorporation of nitrogen as high as 2×10^{18} cm^{-3} has been achieved in CdTe[1] but the highest reported[2] free-hole concentration has been limited to $N_A - N_D \sim 1 \times 10^{17}$ cm^{-3}. In ZnSe, a similar result has been obtained. Incorporation of nitrogen in ZnSe as high as 1.5×10^{20} cm^{-3} has been reported,[3] but activation of carriers is still limited to $N_A - N_D \sim 1.2 \times 10^{18}$ cm^{-3} as measured using capacitance-voltage spectroscopy.[4] Attempts to characterize and understand the nature of the compensating centers in these two cases have been the focus of many recent studies. One important factor is the nitrogen source. The characteristics of the nitrogen source (i.e., ratio of charged to neutral species) influence both the nitrogen incorporation rate and the types of centers which may be produced in epilayers. Recent studies indicate high activation of nitrogen acceptors when ion content of the flux is low.[4]

We have performed a study of ZnSe:N and CdTe:N grown by molecular beam epitaxy (MBE) using two

different sources: one was an Oxford CARS-25 radio frequency (rf) plasma source and the other was an EPI Unibulb rf plasma source. The EPI source, in contrast to the Oxford rf source, has a very high efficiency for producing atomic nitrogen (~70%) while maintaining a very low total ion content. The remaining ions are also expected to be low energy (<10 eV). Both sources gave a high incorporation of nitrogen, as indicated by secondary ion mass spectroscopy (SIMS); however, Hall measurements at room temperature indicate that compensation is still a problem in p-type doping of these materials. We have used liquid-helium-temperature photoluminescence (PL) to monitor changes in the defect structure produced using different source operating parameters. The presence of several impurities in addition to nitrogen can be detected in the epilayers. The concentration dependence of these additional impurities on source-operating parameters suggests considerable source outgassing occurs at the higher rf powers investigated in our growth studies.

EXPERIMENTAL DETAILS

The ZnSe and CdTe epilayers were grown in an MBE system which has been described elsewhere.[5-7] Two rf plasma sources were used in this study of nitrogen doping: the cryogenically cooled CARS-25

Table I. Comparison of Oxford and EPI rf Plasma Sources Used in Nitrogen Doping

Source	Aperture # of Holes-Diameter	Ion Content (%)	Max. Ion Energy (eV)	Atom Efficiency (%) (a)
	1 - 3.0 mm	5	>65	(b)
	9 - 1.0 mm	0.1	40–50	5-6
Oxford CARS	37 - 0.5 mm	0.05	15–25	3–4
	255 - 0.2 mm	0.03	8–12	2–3
EPI Unibulb	400 - 0.2 mm	0.01	(b)	70 (c)

Notes: (a) Atomic N out/molecular N into source x 100% [at 600 W, 6 sccm, by mass spectroscopy]; (b) not measured; (c) projected from GaN growth rate increase at 600 W, 1 sccm flow.

Table II. Source Operating Parameters and Nitrogen Incorporation Data for ZnSe:N

Sample	Source	rf Power (W)	[N] (cm^{-3})
A	Oxford	150	not measured
B	Oxford	200	1.5×10^{19}
			$(N_A - N_D = 2 \times 10^{16})$
C	Oxford	300	8×10^{18}
D	EPI	200 (LB)	8×10^{17} (a)
E	EPI	300	3×10^{19} (a)

Notes: Sources operated in high brightness mode except for sample D (LB = low brightness). (a) Expected concentration based on SIMS data from a sample grown under similar conditions

from Oxford Instruments (United Kingdom) which is very similar to the more standard MPD-21 used for most nitrogen-doping research, and the relatively new Unibulb source from EPI Vacuum Products (Minneapolis, MN). As part of a separate study, an extensive mass and optical spectroscopy characterization was performed[8] on these sources, as summarized in Table I.

The aperture on the Oxford source can be easily changed, thus allowing a range of hole diameters and patterns to be investigated. Several interesting results were determined. While rf plasma sources produce considerably fewer ions than other types of plasma sources such as electron cyclotron resonance, a significant ion content (defined as percentage of total flux) was measured. Both the ion content, and perhaps more importantly, the maximum ion energy, could be lowered by decreasing the diameter of the holes in the aperture plate. Thus, we would expect that samples doped using an aperture with 0.5 mm diameter holes would see about twice as many ions with a factor of two higher energy compared to using an aperture with 0.2 mm diameter holes. It has been reported that ions are one mechanism for compensation in ZnSe:N.[9]

The EPI Unibulb source was specifically designed with a large number (400) of small holes in an attempt to produce a high flux with low ion content and low maximum ion energy. As listed in Table I, the EPI source has a relatively low ion content. From experiments at West Virginia University of MBE growth of GaN epilayers, the EPI source apparently is a factor of 15 to 20 times more efficient at producing atomic nitrogen than the Oxford source. Thus, less total nitrogen flux is required to achieve the same doping level, and the ratio of ions to incorporated nitrogen is much smaller using the EPI source. For example, the reduction in total nitrogen flux along with the reduced ion content indicates that the EPI source will produce about 1% of the ions per nitrogen atom compared to the Oxford source using the 37 holes, 0.5 mm diameter aperture plate.

In the present study, we investigated the differences in doping efficiency between the Oxford and EPI sources. The source discussion in the previous paragraph corresponds to operation in the so-called "high-brightness" mode, where most of the plasma light intensity is due to atomic nitrogen transitions. In addition, both of our sources can be run in a "low-brightness" mode. In this mode, the plasma produces only a few percent of the light compared to high-brightness, and emission spectroscopy indicates that excited molecular nitrogen is the primary output species. We also investigated this mode of operation for p-type doping.

The CdTe samples for this study were grown on (211)B and (100) CdTe, and the ZnSe samples were grown on (100) GaAs. After a standard degreasing, the substrates were prepared as described previously[5,6] and were mounted to the heater block with Aquadag. Prior to CdTe film growth, the bulk CdTe substrates underwent a 20 min atomic hydrogen cleaning at 300°C using a commercial thermal source (EPI). The films were deposited at 260°C using a single CdTe source and flux of 5×10^{-7} Torr beam equivalent pressure (BEP). For growth of ZnSe, the GaAs substrates underwent a similar 20 min atomic hydrogen cleaning at 400°C. Once the substrate reached the growth temperature of 300°C, the Zn source was opened 10 s prior to the opening of the Se source, and BEP readings were 4×10^{-7} Torr for Zn and ~8×10^{-7} Torr for Se. A 0.3 micron thick undoped buffer layer was grown prior to the doped layer, which varied from 1 to 2 microns in thickness.

In the present study, we use PL to evaluate the differences in defect structure for five different ZnSe:N

Table III. Source Operating Parameters and Nitrogen Incorporation Data for CdTe:N

Sample	Source	rf Power (W)	Sibstrate Orientation	Growth Temp (°C)	[N] or $N_A - N_D$ (cm^{-3})
				260	$[N] = 3 \times 10^{16}$
					$N_A - N_D \sim 3 \times 10^{16}$ (b)
G	EPI	200 (LB)	(211)B	260	$[N] = 8 \times 10^{15}$
H	EPI	300	(211)B	260	$[N] = 6 \times 10^{18}$
					$N_A - N_D = 2 \times 10^{17}$
	Oxford	275	(100)	260	$N_A - N_D = 1.5 \times 10^{18}$ (a)
J	Oxford	275	(100)	200	$N_A - N_D = 1.0 \times 10^{17}$ (a)

Notes: Sources operated in high brightness mode, except for samples F and G (LB = low brightness). (a) From a prior study,[7] it was shown that these hole concentrations were apparently due to a large N accumulation at the interface between the doped and undoped layer, and not due to uniform doping; (b) obtained from a CdTe:N film grown using 300 W, low-brightness mode on (100) GaAs.
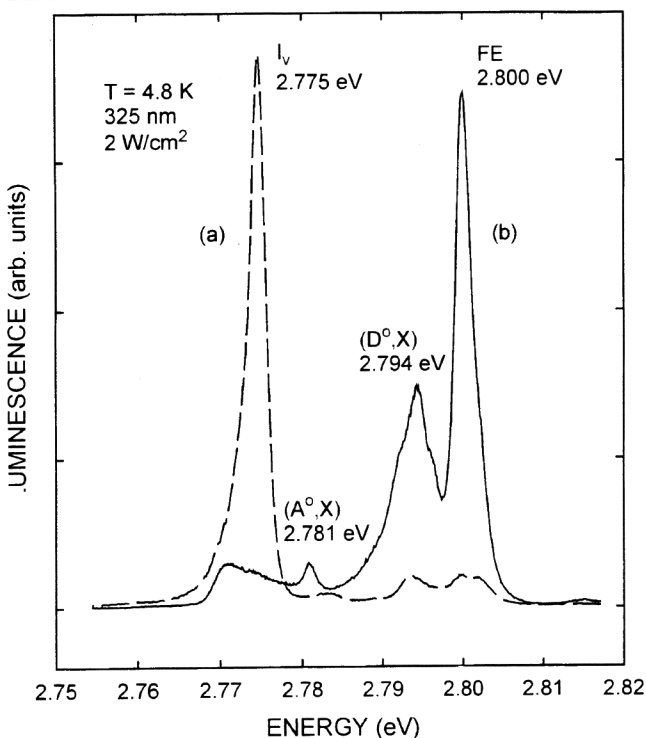

Fig. 1. Excitonic PL at 4.8K from (a) a ZnSe epilayer grown without a Zn pretreatment of the substrate, and (b) a ZnSe epilayer grown with a Zn pretreatment.
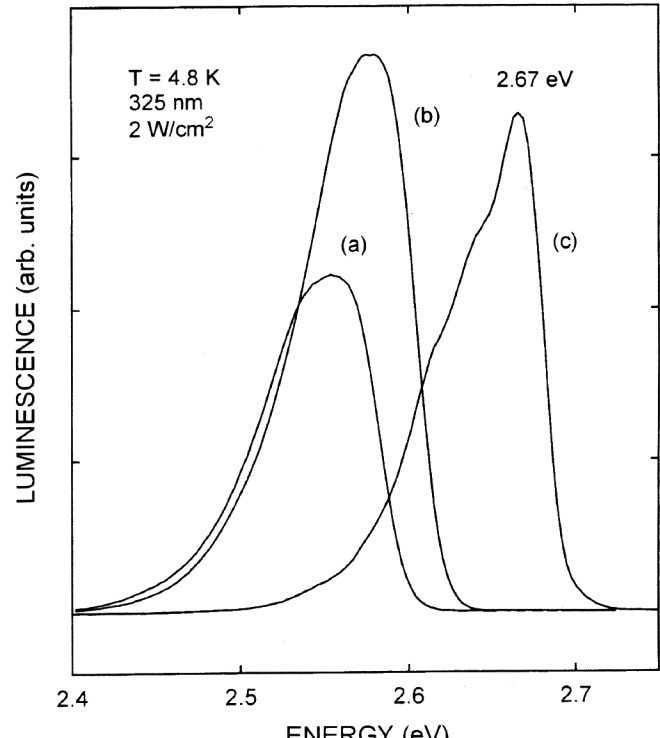

Fig. 2. Photoluminescence at 4.8K from ZnSe:N doped using the Oxford CARS source at different operating powers: (a) Sample A (150 Watts), (b) Sample B (200 Watts), and (c) Sample C (300 Watts, and different source aperture).

samples (referred to as Samples A–E). The nitrogen-rf-source operating parameters used in these film growths are summarized in Table II, along with nitrogen incorporation data. The total N concentration was measured using SIMS, and the free-hole concentration in Sample B was measured at 296K. Five CdTe:N samples (referred to as Samples F–J) were selected for this study, and pertinent data are summarized in Table III for these CdTe films.

Photoluminescence data were obtained from liquid-helium temperatures up to 70K. For ZnSe, excitation was provided by the 325 nm output from a HeCd laser and the incident power density was about 2 W/cm^2. For PL studies of CdTe, the 514.5 nm output from

an argon-ion laser was used for excitation and the power densities were in the range 0.02–0.85 W/cm^2. The PL signals were detected with a grating spectrometer and a photomultiplier tube with GaAs(Cs) cathode. All PL spectra shown in this paper have been corrected for the system response using a calibrated white-light source.

RESULTS AND DISCUSSION

Zinc Selenide

The exposure of the GaAs substrates to a Zn flux prior to the Se flux resulted in a lower defect density in the epilayer. We have also observed that the exci-
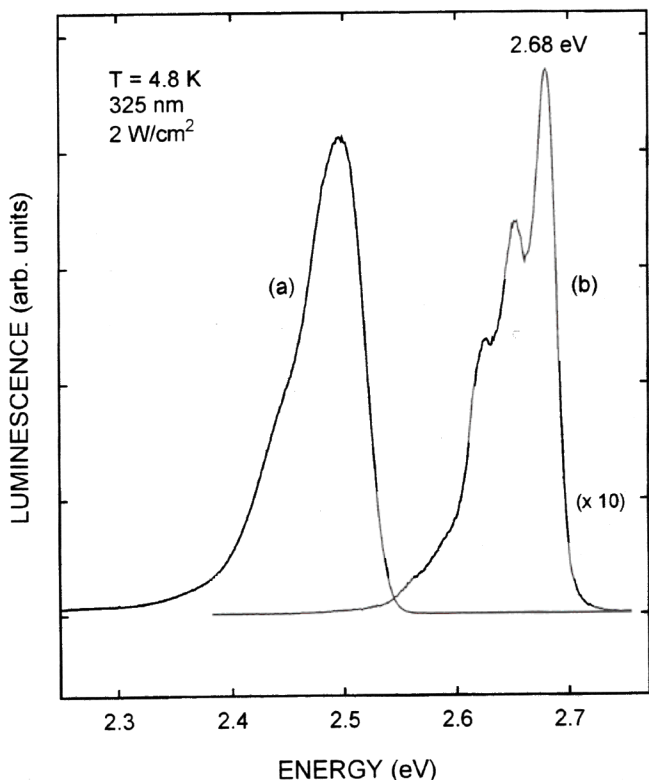
Fig. 3. Photoluminescence at 4.8K from ZnSe:N epilayers doped using the EPI source: (a) Sample E (high-brightness mode), and (b) Sample D (low-brightness mode).
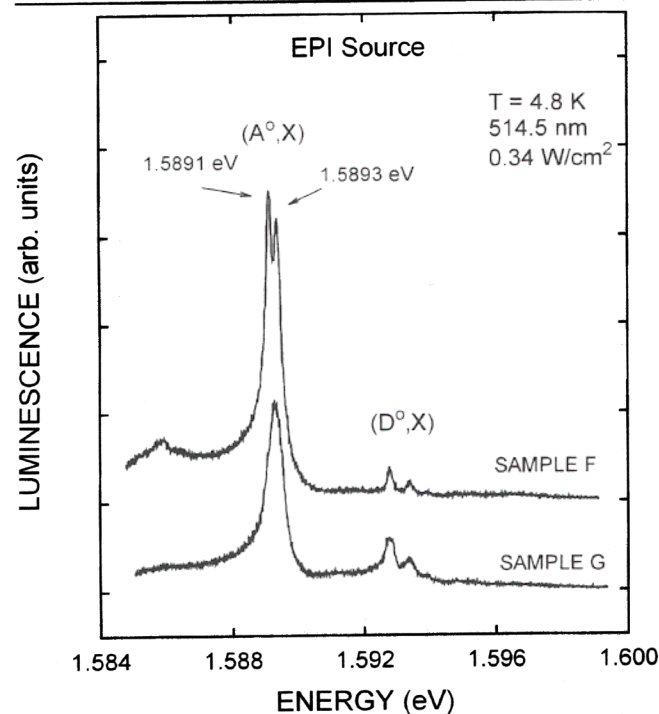

Fig. 4. Excitonic PL at 4.8K from CdTe:N epilayers doped using the EPI source.

using the Zn pretreatment. The sharp bound exciton line at 2.775 eV in curve (a) is often called the I_y line, and has been attributed to Se-site-related defects.[10,11] The Zn pretreatment results in a significant reduction of the I_y line intensity in curve (b). At higher energies, emission lines related to bound-exciton recombination at shallow donors (D^0, X) and free exciton recombination (FE) are shown greatly enhanced in curve (b). Based on these results, the Zn pretreatment was used in subsequent growths of ZnSe:N.

We first describe the PL results obtained from films doped using the Oxford CARS-25 rf plasma source. Representative PL at 4.8K are shown in Fig. 2 for Samples A, B, and C. Samples A and B were grown using rf powers of 150 and 200 W, respectively, and an aperture with 37 holes of 0.5 mm diameter. Both samples exhibit a broad luminescence band shifted in energy below the typical donor-acceptor pair (DAP) emission region. This band is characteristic of heavily doped and highly compensated ZnSe:N epilayers.[3,12,13] Sample C was grown using 300 W and an aperture with nine holes of 0.2 mm diameter (this aperture plate is not included in Table I). The PL from Sample C peaks near 2.67 eV and is attributed to DAP recombination associated with deep donors $(E_D \sim 45–50$ meV) and nitrogen acceptors.[14–17] Sample C has a high concentration of nitrogen (see Table II); however, the smaller aperture-hole diameter, and thus lower ion flux and lower ion energy, has resulted in the typical DAP emission rather than the lower-energy band at ~2.58 eV. We believe the recombination centers involved in the broad-band PL from Samples A and B are of complex origin and are not simply DAP transitions as inferred in earlier work.[12] Our PL results suggested that a nitrogen source with even lower ion flux than the Oxford, such as the EPI Unibulb, may result in even less compensation.

Representative PL spectra for ZnSe samples doped with nitrogen using the EPI Source are shown in Fig. 3. Curve (a) is the luminescence obtained from the heavily doped Sample E grown using the high-brightness mode. Curve (b) was measured from Sample D, which was grown using the low-brightness mode, and exhibits the typical DAP recombination with deep donors. The emission from Sample E is similar to the PL reported in Ref. 3 for a heavily doped sample containing 1.5×10^{20} cm^{-3}. The total N concentration in our sample is 3×10^{19} cm^{-3}. Because of the lower ion content of the EPI source, the shift in PL emission cannot be entirely associated with the presence of nitrogen ions. The broad PL band shifted to lower energy which is detected from Sample E indicates that an additional compensation mechanism is present with heavy N doping. High incorporation rates of N in ZnSe may cause formation of Se vacancies for charge compensation, as indicated by recent electron paramagnetic resonance experiments.[18]

Hall-effect measurements were performed using a standard van der Pauw-Hall geometry in a field of 6000 Gauss. In an attempt to obtain ohmic contacts for characterization of the ZnSe, thin heavily doped

tonic luminescence is affected by the Zn pretreatment in undoped layers. Figure 1 shows the near-edge PL spectra at 4.8K for two undoped ZnSe epilayers: Curve (a) is from a sample grown without the Zn pretreatment, and curve (b) is from a sample grown

$(N_A - N_D \sim 2 \times 10^{19}$ cm^{-3}) ZnTe cap layers were deposited on top of the ZnSe. Using a AuCl$_3$ solution, gold was deposited directly on the ZnTe, and then the ZnTe layer was etched away except directly under the contact. This procedure proved non-optimal, and so accurate Hall measurements were not obtained. However, we could determine that samples doped in the low-brightness mode of the EPI source were quite conducting, whereas samples containing large nitrogen concentrations from operation of either the EPI source or the Oxford source in high-brightness mode were highly resistive, indicating significant compensation. Thus, neither source produced a significant difference in doping efficiencies at high nitrogen incorporation.

Cadmium Telluride

We next describe the CdTe:N results obtained using the EPI and Oxford sources. Representative near-edge PL from CdTe:N doped using the EPI source is shown in Fig. 4. The two samples represent film growths on (211)B CdTe using two different rf powers. Sample F was grown using an rf power of 300

W, and Sample G was grown using an rf power of 200 W. The lower rf power gave rise to PL with one (A^0, X) emission at 1.5893 eV due to substitutional nitrogen.[19,20] The higher rf power resulted in an additional bound-exciton line at about 1.5891 eV. This result was duplicated in several samples, all grown at the higher rf power. A preliminary assignment for the impurity responsible for the 1.5891 eV line is phosphorus. The (A^0, X) energy due to P$_{Te}$ in CdTe has been reported as 1.58897 eV.[19,20]

The PL emissions due to DAP and (e, A^0) recombination for samples grown using either the Oxford source or the EPI source are shown in Fig. 5. Figure 5a shows the PL emission obtained from Samples I and J (refer to Table III for sample details). The two N-doped CdTe samples grown on the (100) substrates reveal a sharp (e, A^0) emission at 1.550 eV and DAP emission at 1.543 eV with accompanying phonon sidebands (Fig. 5a). The N$_{Te}$ acceptor energy obtained from the (e, A^0) emission peak is about 56 meV, in agreement with the reported values of 56,[19,20] 55,[21] and 55.3 meV.[22] The PL from Samples F and G are shown
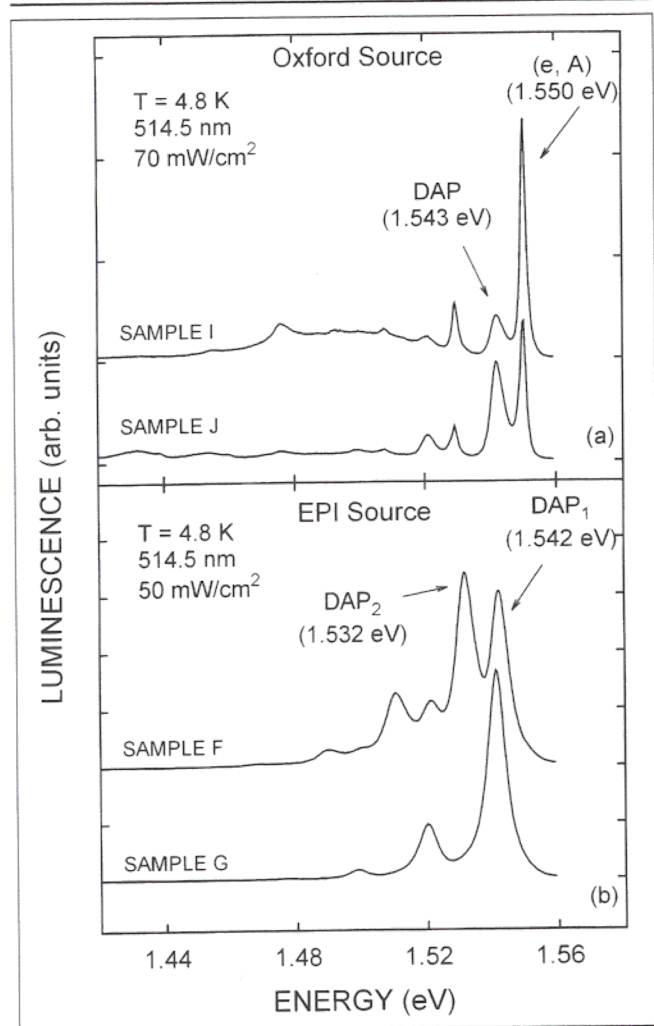

Fig. 5. Donor-acceptor-pair PL at 4.8K from CdTe:N (a) doped using the Oxford source (Samples I and J), and (b) doped using the EPI source (Samples F and G).
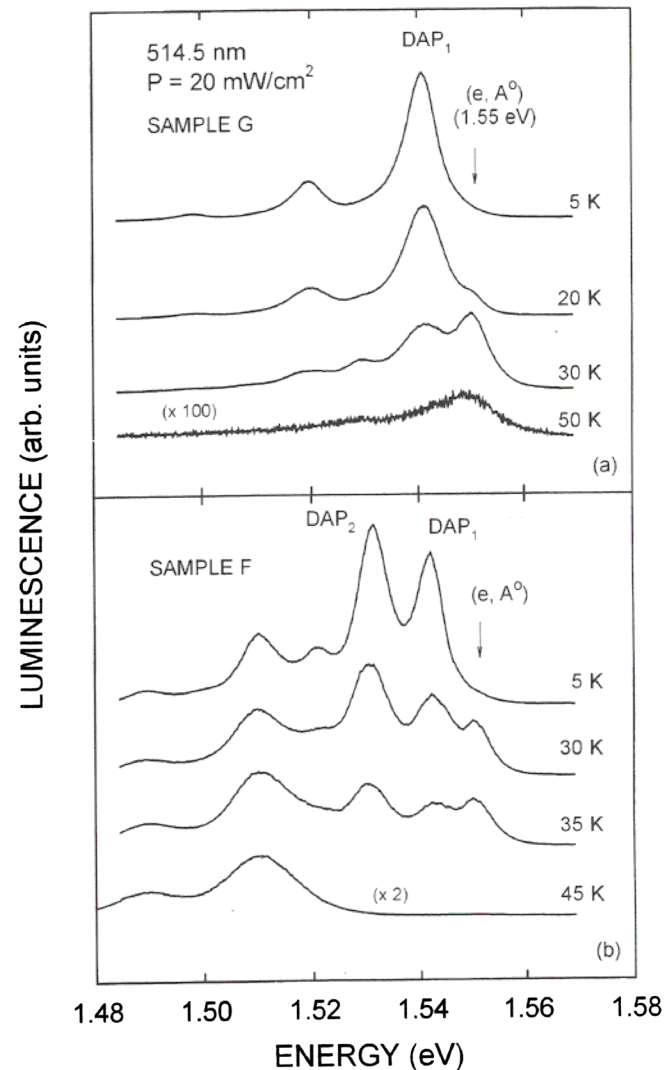

Fig. 6. Temperature dependence of the PL emission from CdTe:N (a) Sample G, and (b) Sample F.

in Fig. 5b. A comparison between Fig. 4 and Fig. 5b shows that Sample G has one (A^0, X) line and one DAP band. In contrast, Sample F has two (A^0, X) lines and two distinct DAP bands labeled DAP_1 and DAP_2 in Fig. 5b. The DAP_1 band is due to nitrogen and a shallow donor. The band DAP_2 occurs at 1.532 eV, so the acceptor impurity responsible for this emission should have an ionization energy of about 66 meV (i.e., 10 meV higher than the N ionization energy). Note that phosphorus is reported to be a 68 meV acceptor in CdTe. Thus, both the DAP_2 energy and the (A^0, X) energy may indicate P as a contaminant arising from the EPI source when operated at higher rf powers.

The (e, A^0) emission was not observed in the PL at 4.8K from the samples doped using the EPI source. The temperature dependence of the PL was thus studied in order to detect (e, A^0) recombination and verify our impurity assignments. The results for Sample G, which exhibited a single DAP band, is shown in Fig. 6a. The DAP band decreased in intensity due to thermal ionization of the shallow donors as the sample temperature is raised. The (e, A^0) band emerges at higher energies and can be seen clearly at 1.55 eV for T > 25K. The PL data for Sample F is shown in Fig. 6b. Again, as the sample temperature is raised, emission related to shallow donors is thermally quenched. Both the DAP_1 and DAP_2 bands decrease in intensity and the (e, A^0) emission due to nitrogen was detected at 1.55 eV at T > 25K. We had hoped to verify the second acceptor energy by detecting a second (e, A^0) emission, but it was not possible to resolve any additional spectral features emerging on top of the DAP_1 band. The temperature-dependence study did, however, reveal an additional emission band at 1.51 eV. This emission band was tracked to 70K. We investigated several other films grown under the higher rf powers and all exhibited a 1.51 eV band, in addition to the second DAP_2 band. Note the absence of the 1.51 eV band in Fig. 6a which corresponds to growth using the lower rf power.

The center responsible for the 1.51 eV band is not known at this time. An emission attributed to a 29 meV donor at 1.52 eV was reported for CdTe:N grown using an electron cyclotron resonance plasma source.[23] The intensity of the 1.52 eV emission increased with increasing ECR power and was believed to be involved in the compensation of nitrogen acceptors. The band at 1.51 eV in Fig. 6b may be similar in origin, and the energy shift due to concentration of compensating centers.

Gold contacts were deposited directly on the CdTe:N samples using the $AuCl_3$ solution. This procedure yields electrical contacts exhibiting ohmic behavior to temperatures as low as 180K. Hall measurements on samples doped using the EPI source in the low-brightness mode indicated 296K hole concentrations of about 3×10^{16} cm^{-3}. These concentration levels were comparable to the nitrogen concentrations measured by SIMS at Charles Evans and Associates (Redwood City, CA), and thus indicate a high degree of dopant activation. The temperature dependence of the measured hole concentration indicated an acceptor activation energy of 56 ± 3 meV, in close agreement with previous studies of nitrogen. In contrast, Hall measurements from a CdTe:N sample doped in the high-brightness mode (Sample H) gave a room temperature $N_A - N_D$ value of about 2×10^{17} cm^{-3}. When compared to the nitrogen concentration of about 6×10^{18} cm^{-3} (as determined by SIMS) in the same sample, greater than 90% compensation is indicated. Similar $N_A - N_D$ values were estimated for samples doped under identical conditions. In the previous study[5,7] of CdTe using the Oxford source, uniform incorporation at this level could not be obtained.

A high uniform incorporation of nitrogen, up to 6×10^{18} cm^{-3}, could be obtained in CdTe using the EPI source; however, compensation was indicated by Hall measurements $(N_A - N_D \sim 2 \times 10^{17}$ cm$^{-3})$. The PL in Fig. 4 and Fig. 5 indicate that higher rf powers introduce additional impurities into the epilayers. The phosphorus levels were low and could not be detected using SIMS; however, we did obtain data indicating that boron was present. The boron apparently arises due to decomposition of the pyrolytic boron nitride commonly used in the rf sources. Boron levels as high as 8×10^{16} cm^{-3} were measured in films grown using the high-brightness mode. Since boron is a Group-III impurity and may be incorporated into the host II-VI lattice as a donor center, it may play a role in compensation of p-type doping. We have also detected the presence of boron in ZnSe:N, grown using the EPI source. The levels of boron indicated by our study, however, are at least an order of magnitude less than the nitrogen incorporation levels, so we do not believe boron is the major compensation center in either CdTe:N or ZnSe:N. Source outgassing is an issue that has not been widely addressed and future studies should focus on the role that rf sources play in the compensation of nitrogen-doped ZnSe and CdTe.

SUMMARY

Nitrogen doping of ZnSe and CdTe epilayers grown by MBE was achieved using two different rf plasma sources: an Oxford CARS-25 and an EPI Unibulb source. We used PL spectroscopy to monitor changes in defect structure and we saw that source outgassing due to decomposition of the PBN can introduce additional defects into the growing layers. Although the EPI source produced a lower ratio of ions to atomic nitrogen, layers doped with large nitrogen concentrations ($\sim 10^{19}$ cm^{-3}) were as heavily compensated as similar layers grown using other rf sources. This implies that the compensation mechanism at this level of nitrogen incorporation is not related to nitrogen ions, even if the role of ions has been shown to be important at intermediate doping levels (i.e., between 10^{17} and 10^{18} cm^{-3}).

ACKNOWLEDGMENTS

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